

4-(4-Chlorophenyl)-3-(2,5-dioxo-2,5-dihydro-pyrrol-1-yl)-1-(4-methoxyphenyl)azetidin-2-one

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Key indicators

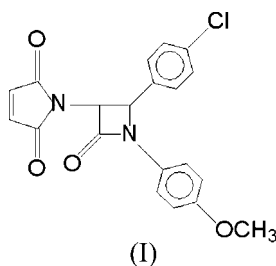
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.038
 wR factor = 0.098
Data-to-parameter ratio = 13.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

There is a planar four-membered β -lactam ring in the title compound, $\text{C}_{20}\text{H}_{15}\text{ClN}_2\text{O}_4$, and the maleimidyl and 4-chlorophenyl groups attached to the β -lactam ring are in *cis* positions.

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Comment

In recent years, research on the synthesis of monocyclic β -lactam derivatives has attracted the attention of chemists (Mandic & Kovacevic, 1996; Li *et al.*, 2001). A general method of preparing a β -lactam is the cycloaddition of a ketene to an imine. During our efforts to synthesize monocyclic lactams using 2-*t*-butoxy-*N*-glyciny succinimide as the source of the ketene, we obtained the title compound, (I), accidentally; the C—O bond of the *t*-butoxy group was cleaved and then elimination of water yielded the double bond. The reason for the cleavage and elimination may be the higher concentration of HCl in SOCl_2 during the reaction of 2-*t*-butoxy-*N*-glyciny succinimide with SOCl_2 (Eder *et al.*, 1977).



A view of the molecular structure is shown in Fig. 1. There is a four-membered ring in the molecule, resulting from the cycloaddition reaction. Atoms N2, C5, C6 and C7 are coplanar with a mean deviation from the plane of 0.0077 Å, and the four bond angles are approximately 90°. The C5—C7 bond [1.568 (3) Å] is longer than a normal C—C bond (1.54 Å), indicating steric hindrance of the substituent groups on C5 and C7. The maleimide and chlorophenyl groups attached to C5 and C7 are directed away from the plane and are in *cis* positions.

Experimental

A mixture of 2-*t*-butoxy-*N*-glyciny succinimide (5 mmol) and SOCl_2 (15 ml) was refluxed for 2 h and then evaporated to obtain the acid chloride. A mixture of 2-*t*-butoxy-*N*-(chloroformyl)methylsuccinimide (5 mmol), *N*-[(4-chlorophenyl)methylene]-4-methoxybenzenamine (3.3 mmol), benzene (30 ml) and Et_3N (1.6 ml) was stirred at room temperature for 6 h. The product was filtered off, washed with 10% HCl and dried over anhydrous Na_2SO_4 . The

benzene solution was evaporated and the residue was purified by column chromatography (ethyl acetate/petroleum ether = 1:3) to give the title product. Single crystals suitable for X-ray analysis were obtained by slow solvent evaporation in air. Analysis calculated for $C_{20}H_{15}ClN_2O_4$: C 62.70, H 0.04, N 0.07%; found: C 62.68, H 0.04, N 0.07%.

Crystal data

$C_{20}H_{15}ClN_2O_4$
 $M_r = 382.79$
 Monoclinic, $P2_1/c$
 $a = 12.344 (3) \text{ \AA}$
 $b = 11.609 (3) \text{ \AA}$
 $c = 13.640 (3) \text{ \AA}$
 $\beta = 112.888 (3)^\circ$
 $V = 1800.7 (8) \text{ \AA}^3$

$Z = 4$
 $D_x = 1.412 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 0.24 \text{ mm}^{-1}$
 $T = 293 (2) \text{ K}$
 Block, yellow
 $0.26 \times 0.22 \times 0.16 \text{ mm}$

Data collection

Bruker APEX-II CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.845, T_{\max} = 0.866$
 (expected range = 0.939–0.962)

9542 measured reflections
 3173 independent reflections
 2008 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$
 $\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.098$
 $S = 1.01$
 3173 reflections
 245 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0178P)^2 + 0.6522P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$

H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C–H distances of 0.93–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C})$.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve

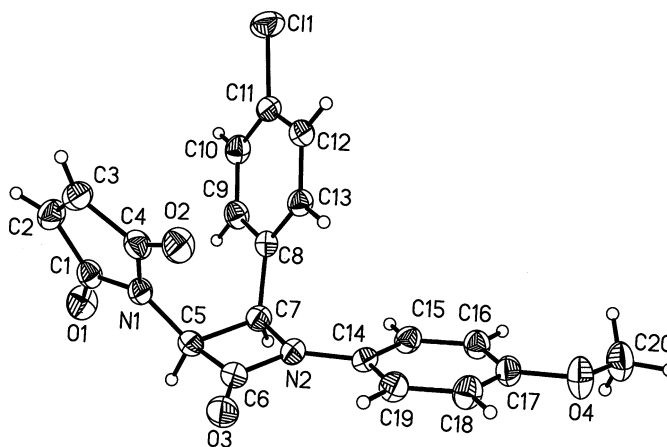


Figure 1

A view of the title compound. Displacement ellipsoids are shown at the 30% probability level.

structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

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